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In re Application of: :  
*Bruce J. Kokko* : Examiner: S. Alvo  
U.S. Serial No. 09/456,270 : Group Art Unit: 1731  
Filed: December 7, 1999 :  
Docket No. 2130 (FJ-99-12) :  
For: METHOD OF MAKING ABSORBENT :  
SHEET FROM RECYCLE FURNISH :  
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Mail Stop RCE  
Commissioner for Patents  
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Alexandria, Virginia 22313-1450

THIRD DECLARATION OF BRUCE J. KOKKO

Bruce J. Kokko, inventor of the subject matter of the above-noted patent application hereby declares:

1. That he was awarded a Ph.D. degree in Chemistry from the University of Illinois, Urbana, Illinois, in 1983. Since that time he has worked on projects involving chemistry relevant to the manufacture of paper, including sheet useful for making paper towel and paper tissue.
2. That the invention of the above-noted patent application resides, in part, in the discovery that nonionic surfactants having specific HLB values can be combined with quaternary surfactants to make debonding compositions exhibiting unexpectedly enhanced ability to provide tensile reduction in absorbent sheet manufacture. The invention thus enables the use of less quaternary surfactant to achieve a given tensile reduction, for example, or enables more tensile reduction with a given amount of quaternary surfactant. These features are particularly

significant in the manufacture of absorbent sheet from recycle furnish and/or where the amount of cationic charge addition which can be tolerated in a papermaking process is limited. Oftentimes the addition of cationic species such as quaternary surfactants is limited in a papermaking machine as a practical matter by operating problems such as plate-out when the cationic content goes too high. Thus, prior to the invention of this application, there was a significant need to reduce quaternary surfactant levels required for a given level of tensile reduction. As can be seen from the data which follows, such results are readily achieved by way of the invention.

3. That he understands that the data submitted in November, 2002 in this application was deemed insufficient to place this application in condition for allowance because there was no direct comparison of a composition of the invention based on methylimidazolinium quaternary surfactant and PEG dioleate which was a "species" of the invention elected for examination and which components are used in Example Series A (the formulation used in series A being prior art for present purposes) and in Example O of the specification (an embodiment of the claimed invention). Following, the data submitted in November, 2002 is supplemented with tensile results achieved with Formula O, which is a methylimidazolinium/PEG dioleate composition wherein the PEG dioleate has a higher HLB value than the dioleate of Example Series A because it has a longer ethylene glycol chain. Details appear in Paragraph 4 below. Note that the PEG dioleates of Example Series A and Example O are different in that they have different HLB values, but the debonder formulations in both cases are methylimidazolinium quat/ PEG dioleate compositions.
4. That following the procedure of Example 1 of the application, absorbent sheet was prepared: (a) without debonder; (b) with the debonder of Example Series A at an add-on rate of 6 lbs per ton of fiber; (c) with the debonder of Formula O of the application at an add-on rate of 6 lbs per ton of fiber; and (d) with the debonder of Formula P of the application at an add-on rate of 6 lbs per ton of fiber. The quaternary and nonionic surfactant content of Example Series A, Formula O and Formula P are set forth below:

Formulation A: 75 wt.% of a mixture of 1-(2-octadecenamidoethyl)-2-heptadecenyl-3-methylimidazolinium methylsulfate, 1-(2-octadecenamidoethyl)-2-heptadecenylimidazoline and 10 wt.% PEG-6-dioleate and 10 wt.% PEG-6-2-tridecanol.

Formula O: 38 wt% mixture of 1-(2-octadecenamidoethyl)-2-heptadecenyl-3-methylimidazolinium methylsulfate and 1-(2-octadecenamidoethyl)-2-heptadecenylimidazoline, 50 wt% PEG-600-dioleate, 7.3 wt% PEG-400-monolaurate, 3.8 wt% propylene glycol, and 0.8 wt% methylolate.

Formula P: 44.5 wt% mixture of 1-(2-octadecenamidoethyl)-2-heptadecenyl-3-methylimidazolinium methylsulfate and 1-(2-octadecenamidoethyl)-2-heptadecenylimidazoline, 50 wt% PEG-400-monooleate.

Results of tensile tests on samples of the sheet prepared as noted above as well as HLB values appear in the table below:

Debonder Formulation	Dosage		aHLB <sup>1</sup>	Fatty Acid Carbon Chain Length of Nonionic Surfactant	Tensile Strength (km) <sup>2</sup>	% Reduction <sup>3</sup>
	Add-On Rate(#/T)	mol Quat/T				
Control	NA	NA	NA	NA	2.7	NA
Formulation of Example Series A	6	1.8	9.8	18/13	2.1	23
Formulation of Example O	6	1.2	10.7	18/12	1.97	27
Formulation of Example P	6	1.4	11.8	18	1.7	37

1) Actual HLB of nonionic surfactant(s) fraction of product.

2) Dry Breaking Length (dry tensile normalized for basis weight).

3) Percent reduction in dry breaking length relative to control.

- That results shown in the above Table show unexpectedly large tensile reductions utilizing the invention as embodied in the formulations of Example O and P as opposed to Example Series A. The formulation of Example Series O has 1/3 less quaternary surfactant than the Example Series A composition, yet exhibits significantly more tensile reduction at the same add-on levels (4/23 or about 17% more). This is contrary to conventional wisdom wherein it was believed that

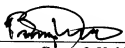
tensile reduction would decrease with decreasing quaternary surfactant usage. One would have expected the formulation of Example O to exhibit significantly less tensile reduction than the formulation of Example Series A and the fact it exhibits more tensile reduction was an unexpected, surprising and very useful result.

6. Likewise, the formulation of Example P would have been expected to exhibit less tensile reduction than the formulation of Example Series A at the same add-on rates because it also contains less quaternary surfactant. The contrary result shown in the above table is again unexpected.
7. That the nonionic surfactant used in Formula O is primarily a PEG dioleate as is the nonionic surfactant used in Example Series A; however, the PEG dioleate surfactant used in Formula O has a higher HLB than the PEG dioleate used in Example Series A because the PEG content is higher. Note that the quaternary surfactants used in Example O and Example Series A are identical. By virtue of its composition, the use of Formula O is within the scope of Claim 1 of the above-noted patent application.
8. That the nonionic surfactant used in Formula P was a PEG-400 monooleate ester (a monoalkylated nonionic surfactant) having a hydrophobic carbon chain length of 18 and an HLB value of 11.8. The use of debonder Formulation P is within the purview of Claim 1, subparagraph (c) by virtue of these characteristics and the presence of the nonionic surfactant in the mixture within the range of from 25 to 60 weight percent based on the total amount of surfactant.
9. That in the Series A experiment detailed above, the nonionic surfactants used were a PEG-6-dioleate ester (a dialkylated nonionic surfactant) having a hydrophobic carbon chain length of 18 and a PEG-6-2 tridecanol (a monoalkylated nonionic surfactant) having a hydrophobic carbon chain length of 13. This nonionic surfactant mixture had an HLB value of 9.8. By virtue of these characteristics, the composition is excluded from Claim 1 of the application which calls for HLB values of greater than 10 for such compounds in the mixture (*see*

Claim 1, subparagraphs a and d). The debonder formulation of Example Series A is also excluded from Claim 1 because it has less than 25 percent by weight nonionic surfactant, specifically, it had 20% by weight nonionic surfactant.

10. The undersigned Declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the subject application or any patent issuing thereon.

Dated 5-10-03

  
Bruce J. Kokko